# Research Update: Physical Sciences Research

# **Solidifying the Future**

Scientists use microgravity, where buoyancy is minimized, to see how bubbles form and move within molten metals and alloys as they solidify.

he beautiful structure of a snowflake is a well-known example of the way tiny water droplets freezing into ice form branching crystals. Such branching crystals are called "dendrites" from an ancient Greek word for tree. As a puddle freezes, however, a continuing dendritic network of ice crystals forming across its surface traps air within the water, bubbles that remain when the puddle is frozen solid.

Many metals and alloys also have dendritic structures. When molten metals or alloys are solidified for commercial applications, uniformly distributing the dendrites and controlling or eliminating gas pockets are crucial to ensuring the materials' strength.

Richard Grugel at Marshall Space Flight Center, investigator for the Microgravity Science Glovebox (MSG) on the International Space Station (ISS), is seeking to understand the subtle forces that act on gas bubbles in molten metals and alloys. Grugel and his team have created the Pore Formation and Mobility Investigation (PFMI) to study how bubbles move and interact with one another as a material is melted and solidified in microgravity.

## **Gravity and Bubbles**

On Earth, when a metal is liquid, gravity-driven buoyancy dominates the movement of any bubbles that form, often causing them to rise quickly to the surface, pop, and disappear. But as a metal cools, bubbles can get trapped between dendrites forming in the bulk of the material or under the solidifying skin on top of a casting. Such bubbles become pores: trapped pockets of gas that diminish the material's usefulness.

Gravity-driven buoyancy so dominates the behavior of bubbles on Earth that it hinders scientists from observing slighter influences on their dynamics — and thus from possibly discovering other clues to the causes of porosity. In microgravity, however, buoyancy is minimized: bubbles do not rise and disappear, allowing for an in-depth study of their subtler behavior.

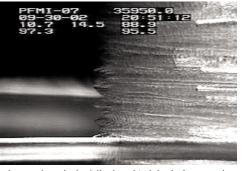
Grugel hopes that data from his PFMI experiment will be useful in designing future microgravity investigations, as several previous materials-processing experiments in space resulted in bubbles trapped within the solid. He also hopes PFMI will provide insight toward understanding the dynamics of bubble interactions during materials processing in Earth's gravity.

#### At Work on the ISS

The PFMI apparatus flew to the ISS with the Expedition 5 crew aboard space shuttle mission STS-111 in June 2002. The PFMI thermal chamber, the experiment cameras, and other data-collecting devices were installed in the MSG, a rack-sized, enclosed working volume that allows astronauts to conduct experiments in a contained environment.

At the heart of the PFMI are 15 samples containing succinonitrile (SCN), a transparent plastic-like material with a low melting point commonly used to model and directly observe melting and solidifying processes that occur in metal alloys. Five of the samples are pure SCN; the other 10 are mixed with less than 1 percent of water to simulate alloys. (An alloy is composed of at least one metal; the samples used in PFMI are better described as a mixture of two compounds, but their behavior is very similar to that of an alloy.) Because water acts as an "alloying" agent, "we will definitely see more dendritic-type structures in these samples," says Grugel.

The samples are preloaded in customized glass tubes 1 cm (0.4 inches) in diameter and 20 cm (7.9 inches) in length.



Taking a closer look at the liquid/solid interface, a network of branching dendrites form into a succinonitrilewater sample like a preliminary skeleton for the solid to take shape. This video image shows tiny bubbles trapped within the crystal branches, a problem sometimes encountered in materials processing on Earth. In its melting and solidifying, the succinonitrile-water sample mimics the behavior of metal alloys.

Running along the inside of the filled tubes are six thin protective metal sheaths of increasing length, each of which contains a thermocouple that continuously measures the temperature in the sample during processing. (A thermocouple is a wire made of two dissimilar metals bonded together; heating induces a voltage difference that is a sensitive indicator of temperature.) Inside the far end of the tube is a compression spring and piston that exert a constant pressure on the sample to eliminate any gaps that might develop as the material expands during melting. Two video cameras record the sample's behavior in the thermal chamber during each 10- to 12hour experiment run.

Similar to the commercial process for making turbine blades, PFMI uses a process called controlled directional solidification. As each experiment begins, a heating and cooling mechanism that surrounds the glass tube slowly moves along it in one direction and heats the sample to a maximum of 130 °C (266 °F). Pure SCN melts at 58 °C (136 °F) and the SCN-water compound melts at about 55 °C (131°F); the extra heat is used to establish temperature gradients along the sample. As the sample is heated, the formation of bubbles and their behavior within the melt are observed and recorded. After the melting cycle is completed, the surrounding mechanism begins its cooling cycle and the direction of

its movement along the tube is reversed. Thus the molten sample is re-solidified under controlled conditions. "During resolidification of the SCN-water mixtures, aligned dendrites form and we have observed bubbles generating between them," says Grugel.

mechanical properties. Collectively, the dendrites are essentially a framework for the solidifying material, influencing its structural strength. "Hopefully, the experiment's data will be useful in mathematical models, with the intent of eventually being able to tailor specific material properties

during solidification," says
Grugel.

The temperature gradient in the molten material ahead of the solidifying material also affects the movement of bubbles. With buoyancy effects minimized in microgravity, bubbles might not be expected to move through the liquid. However, any subtle effects acting on a bubble that are normally masked by Earth's gravity are revealed in orbit.

"Our experiment is set up so that for a few cen-

timeters the temperature of the liquid increases as it moves away from the solid," explains Grugel. "Thus, when a bubble is released from the solid, its leading side into the liquid is slightly warmer than its rear." The imposed temperature gradient across the bubble affects molecules on its surface and causes a flow on the bubble's surface that usually moves the bubble from cooler to warmer temperatures, something that buoyancy forces overwhelm in Earth's gravity.

"We have directly observed that when bubbles leave the solid and move up the temperature gradient, they stop when the temperature of the surrounding liquid is the same as that of the bubble," says Grugel. "These results, obtained under dynamic conditions, could only be observed in microgravity" and should be highly interesting when compared with theory. Grugel believes the results could be useful to materials processing both in microgravity and on Earth.

#### Results to Build On

The first PFMI experiment in microgravity was conducted in September 2002. Grugel and his team of investigators occupied the Telescience Center (TSC) at Marshall's Microgravity Development Laboratory, watching snatches of video from the experiment every 5 to 15 minutes. When the ISS was above the horizon, they also sent commands to the experiment, changing the temperature, growth rate, and other variables.

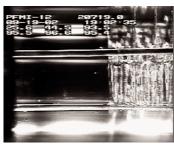
"The video downlink from the station during the runs was essential to conducting the experiments," says Grugel. "We are very pleased with the results. The entire set of real-time videotapes for the completed samples were brought back with STS-113 in December 2002, and is being analyzed."

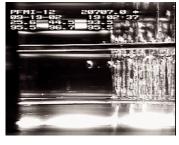
Between September and December, eight samples were tested, two experiment runs prematurely ending about halfway through processing due to anomalies (one a "single event upset" in the PFMI motor control that was quickly restored, the other when the MSG lost power). Still, Grugel says he is pleased with the MSG as a research facility. The MSG has well demonstrated its capability, and Grugel believes its generic design makes it a potential candidate for future investigations. "For relatively low cost, we are getting good value out of the glovebox," Grugel says. "The station crew has been very accommodating. Also, I cannot give enough credit to our small but dedicated and talented ground-based team."

Grugel reports that he and his colleagues are evaluating the data they have so far to get quantitative measurements and compare the findings with current theoretical models. "I suspect that upon careful examination of the tapes we will get more than we expected about the dynamics of bubbles and solidification under controlled processing conditions in microgravity," says Grugel.

Chris McLemore

For more information about the PFMI study go to http://www1.msfc.nasa.gov/NEWSROOM/background/ facts/PFMI.html.









This series of photographs from an International Space Station experiment conducted in September 2002 shows melting solid pure succinonitrile (on the right of each image) and bubbles being released into the liquid (on the left). According to theory, two prime factors determining bubble movement are the temperature gradient in the liquid ahead of the solid and the size of the bubble. The numbers in the top left corner represent (from left to right) sample number, position, date, time, and the six temperatures collected from the thermocouples, which are the horizontal lines.

### Various Variables

Grugel explains that there are three main processing variables: "alloy composition, the growth velocity of the solid, and the temperature gradient in the molten material ahead of the solid." How those variables affect the solid's microstructure is essential because they are what determine the material's strength and other properties. For PFMI, Grugel is primarily concerned with the growth velocity and the temperature gradient.

"The growth velocity," explains Grugel, "is the rate at which the molten material solidifies." A minimum growth velocity is needed for dendrites to form; moreover, different growth velocities create specific dendritic patterns. The faster the molten material solidifies, the finer the dendritic pattern that results. A finer microstructure generally yields better